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Indian Standard

SPECIFICATION FOR COCOA MASS

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Indian Standard

SPECIFICATION FOR COCOA MASS

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Indian Standard

SPECIFICATION FOR COCOA MASS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 15 December 1986, after the draft finalized by the Stimulant Foods Sectional Committee, had been approved by the Agricultural and Food Products Division Council.

0.2 Quality estimation of 'Cocoa mass' assumes importance, because it is marketed as a finished product and is used as a major ingredient/raw material in the manufacture of chocolate, chocolate coating, cocoa beverages, bakery goods and confectionery.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value of this standard.

1. SCOPE

1.1 This standard prescribes the requirements and method of sampling and test for cocoa mass.

2. REQUIREMENTS

2.1 Description — Cocoa mass shall be a solid or semisolid material prepared by adequate grinding of commercially shell free nibs (cotyledons) of well fermented, dried and roasted cocoa beans, the seeds of *Theobroma cacoa.*

2.2 The product shall have its characteristic colour, odour and flavour and it shall be free from any added colouring matter, flavour, preservative, adulterants, harmful ingredients, or added fats other than Cocoa butter.

*Rules for rounding off numerical values (*revised*).

2.3 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR COCOA MASS

(*Clauses* 2.1 *and* 5.1)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Appendix) of this Standard	Related Indian Standard
(1)	(2)	(3)	(4)	(5)
i)	Moisture, percent *m/m*, *Max*	3	A	—
ii)	Fat, percent *m/m*, moisture-free basis, *Min*	47	B	—
iii)	*p*H	5 ± 0·5		
iv)	Total ash, percent *m/m*, moisture and fat free basis, *Max*	8·0	C	—
v)	Alkalinity of ash, percent *m/m*, moisture & fat free basis, *Max*	5·0	D	—
vi)	Acid insoluble ash, percent *m/m* moisture & fat free basis, *Max*	0·2	E	—
vii)	Crude fibre, percent *m/m*, moisture & fat free basis, *Max*	7·0	F	—
viii)	Particle size, percent material passing through 150 mesh, *Min* (wet sieving)	99	G	—
ix)	Standard plate count of bacteria per g, *Max*	50 000	—	IS : 5402-1969*
x)	Yeast and mould per g, *Max*	100	—	IS : 5403-1969†
xi)	E coli per g, *Max*	Nil/g	—	IS : 5401-1969‡

*Method for plate count of bacteria in foodstuffs.
†Method for yeast and mould count in foodstuffs.
‡Methods for detection and estimation of coliform bacteria in foodstuffs.

2.4 Optional Treatment and Ingredients — The material may at any stage during its manufacture be treated with alkalizing agents, such as hydroxides, carbonates and bicarbonates of sodium, potassium, magnesium and aluminium only not exceeding 5 percent singly or in combination and expressed as anhydrous potassium carbonate on fat free basis. The maximum level of neutralizing agents used, namely, Citric, tartaric or phosphoric acid shall be 0·5 percent in the final product. For alkalized Cocoa mass, a) total ash (on moisture and fat free basis) percent m/m Max 14% and b) alkalinity of ash (on moisture and fat free basis), percent *m/m Max* 7%, c) *p*H 6 ± 0·5.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in clean, sound and odour free containers made of tin plate, plastic, grease proof paper and aluminium foil, as agreed to between the purchaser and the vendor. The net content packed in each container shall be 500 g or more.

3.2 Marking — The following particulars shall be marked or labelled legibly and indelibly on each container.

a) Name of the material,
b) Name of the manufacturer,
c) Batch or code number,
d) Date of manufacturing, and
e) Net mass of contents.

3.2.1 Each container may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 The method of drawing representative sample of the material and the criteria for conformity shall be as prescribed in Appendix H.

5. TESTS

5.1 Tests shall be carried out as prescribed in Col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities that affect the results of analysis.

*Specification for water for general laboratory use (*second revision*).

APPENDIX A

[*Table* 1, *Item* (i)]

DETERMINATION OF MOISTURE

A-1. PROCEDURE

A-1.1 Preparation of Sample — Cool the material until hard and then grate or shear to a fine granular condition.

A-1.2 Vacuum Oven Method

A-1.2.1 Weigh accurately about 10 g of the prepared sample (*see* **A-1.1**) in a tared weighing bottle having a diameter of about 40 mm and a height of about 25 mm. Distribute the material as evenly as possible over the bottom of the bottle by gentle tapping. Place the bottle in a vaccum, oven remove the cover of the bottle and dry the material for 6 hours at 80 ± 1°C at a pressure not exceeding 5 mm of mercury. Allow the bottle to cool to room temperature and weigh.

A-1.3 Air Oven Method

A-1.3.1 Weigh accurately about 2 g of the sample (*see* **A-1.1**) in a petridish tared with its cover. Distribute the material as evenly as possible over the bottom of the petridish by gentle tapping. Place the dish in an oven set at 105 ± 2°C. Remove the cover and place it next to the dish. Dry for 3 hours and cool in a desiccator for about 20 minutes. Weigh and repeat drying for 30 minutes intervals till the difference between two successive weighing is not more than 1 mg.

A-2. CALCULATION

A-2.1 Moisture, percent $m/m = \frac{100\ (M_1 - M_2)}{M_1}$

where

M_1 = mass in g of the prepared sample taken for the test, and

M_2 = mass in g of the material after drying.

APPENDIX B

[*Table* 1, *Item* (ii)]

DETERMINATION OF FAT

B-1. PRINCIPLE

B-1.1 The fat in cocoa mass is enclosed by cellulose. It is liberated by digesting with hydrochloric acid. The digested material is filtered in a suitable manner to remove the acid solution, it is dried and the fat is then extracted in a soxlet apparatus, and the residual fat in the flask dried and weighed.

B-2. REAGENTS

B-2.1 Hydrochloric acid, chemically pure, 25% by weight (Sp Gr 1·12) (*see* IS : 265-1976*).

B-2.2 Petroleum ether, dried, freshly distilled, boiling point below 60°C.

B-2.3 0·1 N Silver Nitrate solution ($AgNO_3$) (*see* IS : 2214-1977†).

B-3. PROCEDURE

B-3.1 Digesting the Sample — Weigh 3 to 4 g of sample prepared in **A-1.1** in a 500 ml beaker accurately. Add 45 ml of boiling hot distilled water into the beaker and stir to give a homogenous suspension. Again stirring continuously, add 55 ml of 25% hydrochloric acid (giving 4 NHCL). Add a few defatted, incinerated pieces of pumice stone or pumice powder. Cover the beaker with a watch glass or connect it to a reflux condenser in order to avoid losses by splashing and to prevent the acid from becoming too concentrated due to evaporation of the water. Bring the contents of the beaker slowly to boiling point. When boiling starts, remove the flame momentarily to avoid overflowing. Boil the contents gently for about 15 minutes. Rinse watch glass or condenser used above into the beaker with 100 ml of boiling water. Filter the digest while still hot through a wetted, fat free, fluted filter paper of such a pove size as to allow the filtration to proceed at a reasonable speed. Wash the beaker several times with hot water and also pass the washings through the filter paper. Wash the filter paper with several further lots of hot water until the filtrate ceases to give a chloride reaction with silver nitrate. While still wet, transfer the filter paper with sample to a defatted extraction thimble and dry in a small beaker for up to 6 hours at 100-101°C.

B-3.2 Extraction — Place a few pieces of pumice stone into a 250 ml flat bottomed soxhlet flask and dry for 1 hour in an oven at 100-101°C.

*Specification for hydrochloric acid (*second revision*).

†Specification for silver nitrate, pure and analytical reagent (*first revision*).

Cool the flask in a desiccator for 30 minutes and accurately weigh on an analytical balance.

Place the thimble containing the dried filter paper with the digested sample into a soxhlet extractor. Rinse the beaker (dried) which was used for the digestion several times with petroleum ether and pour the washings into the thimble with soxhlet extractor.

Extract the digested sample and the filter paper under a reflux condenser for 4 hours using 40-50 ml petroleum ether. After completion, distil off the petroleum ether on a water bath and dry the flask with the fat, lying, on its side, either under vacuum at 70°C, or in on an oven at 100-101°C. After drying, remove the last traces of ether by blowing air into the flask using a rubber baloon. Cool the flask for 30 minutes in a desiccator at room temperature. Reweigh on an analytical balance.

B-4. CALCULATION

B-4.1 Fat (on moisture free basis), percent $m/m = \frac{10\,000 \times A}{M_1 \times S}$

where

A = extracted fat in the flask in g,
M_1 = mass of sample in g, and
S = percent dry matter in sample.

APPENDIX C

[*Table* 1, *Item* (iv)]

DETERMINATION OF TOTAL ASH

C-1. PROCEDURE

C-1.1 Weigh accurately about 10 g of the material in a porcelain dish. Heat at 100°C until water is expelled and then heat slowly over a flame until swelling ceased. Ignite in a muffle furnace at 550°C until grey ash results. Cool in a desiccator and weigh. Repeat the process of igniting, cooling and weighing at half hour intervals until the difference in weight between two successive weighings is less than one milligram.

C-2. CALCULATION

C-2.1 Total ash (on moisture and fat free basis), percent m/m

$$= \frac{10\,000\ m_1}{m_2\,[\,100 - (M + F)\,]}$$

where

m_1 = mass in g of the ash;

m_2 = mass in g of the prepared sample taken for test;

F = fat (on as is basis), percent by mass in the prepared sample; and

M = moisture, percent by mass, in the material (*see* **A-2.1**).

APPENDIX D

[*Table* 1, *Item* (v)]

DETERMINATION OF ALKALINITY OF ASH

D-1. REAGENTS

D-1.1 Dilute Hydrochloric Acid — approximately 0·1 N.

D-1.2 Standard Sodium Hydroxide — approximately 0·1 N, accurately standardized.

D-2. PROCEDURE

D-2.1 Weigh accurately about 2 g of the material, and ash as prescribed in **C-1.1**. Add a known excess of dilute hydrochloric acid and boil for 2 minutes.

Cool and titrate the excess of acid against standard sodium hydrochloride using bromocresol in green as indicator till the colour changes to green.

D-2.1.1 Titrate 10 ml of dilute hydrochloric acid against the standard sodium hydroxide using phenolphthalein as indicator till the colour changes to pink.

D-3. CALCULATION

D-3.1 Alkalinity of ash (as K_2O) (on moisture and fat free basis), percent by mass

$$= \frac{47{\cdot}1\ N\left(\frac{V_2 - V_1 - V_3}{10}\right)}{m\,[\,100 - (M + F)\,]}$$

where

N = normality of standard sodium hydroxide (*see* **D-1.2**),

V_2 = volume in ml of dilute HCl added (*see* **D-2.1**),

V_1 = volume in ml of standard sodium hydroxide corresponding to 10 ml of dilute hydrochloric acid,

V_3 = volume in ml of standard sodium hydroxide required for the excess of acid,

m = mass in g of the material taken for the test (*see* **2.1**),

M = moisture, percent m/m, in the material (*see* **D-2.1**), and

F = fat (Cocoa butter), percent m/m, in the material.

APPENDIX E

[*Table* 1, *Item* (vi)]

DETERMINATION OF ACID INSOLUBLE ASH

E-1. REAGENT

E-1.1 Dilute Hydrocholoric Acid — approximately 5 N, prepared from concentrated hydrochloric acid (*see* IS : 265-1976*).

E-2. PROCEDURE

E-2.1 To the ash contained in the dish (*see* **C-1.1**) add 25 ml of dilute hydrochloric acid, cover with a watch glass and heat on a boiling water bath for 10 minutes. Allow to cool and filter the contents of the dish through Whatman filter paper No. 42 or its equivalent. Wash the filter paper until the washings are free from the acid. Return the filter paper and residue to the dish. Keep it in an electric air oven maintained at 135 $\pm$ 2°C for about 3 hours. Cool the dish in a desiccator and weigh. Repeat the process of igniting in a muffle furnace, cooling and weighing on half hour intervals until the difference in mass between two successive weighings is less than 1 mg. Note the lowest mass.

E-3. CALCULATION

E-3.1 Acid insoluble ash

(on moisture and fat free basis), percent m/m

$$= \frac{10\,000\, m_1}{m_2\,[\,100 - (\,F + M\,)]}$$

where

m_1 = mass in g of the acid insoluble ash;

m_2 = mass in g of the prepared sample taken for the test;

*Specification for hydrochloric acid (*second revision*).

M = moisture, percent by mass in the prepared sample (**A-2.1**); and

F = fat (on as is basis), percent m/m, in the prepared sample.

APPENDIX F

[*Table* 1, *Item* (vii)]

DETERMINATION OF CRUDE FIBRE

F-1. REAGENTS

F-1.1 Ether — solvent grade (*see* IS : 336-1973*).

F-1.2 Ethyl Alcohol — (*see* IS : 321-1964†).

F-1.3 Sodium Oxalate Solution — one percent (*m/v*).

F-1.4 Dilute Sulphuric Acid — 1·25 percent (*m/v*), accurately prepared.

F-1.5 Sodium Hydrochloride Solution — 1·25 percent (*m/v*) accurately prepared.

F-1.6 Asbestos — Gooch-grade, medium fibre, acid-washed and ignited.

F-2. PROCEDURE

F-2.1 Preparation of Moisture and Fat Free Material — Treat 7 g of the prepared sample (**A-1.1**) in a centrifuge bottle twice, with 100 ml of ether. Centrifuge and decant superanatant liquid after each addition of ether. Dry the residue in an air-oven at 100 ± 2°C and powder it with a flattened glass rod. Wash with 100 ml of water centrifuge for 10 minutes and decant the aqueous alcohol twice and with 100 ml of ether once, in the same manner as with water. Transfer the moisture and fat-free residue to a dish and dry it to constant weight in an air-oven at 105°C. Use this for the determination of crude fibre (**F-2.2**).

F-2.2 Weigh accurately above 2·5 g of the residue (**F-2.1**) and transfer to a litre flask. Take 200 ml of dilute sulphuric acid in a beaker and bring to the boil. Transfer the whole of the boiling acid to the flask containing the residue and immediately connect the flask with a reflux water condensor and heat, so that the contents of the flask begin to boil within one minute. Rotate the flask frequently, taking care to keep the

*Specification for ether (*second revision*).

†Specification for absolute alcohol (*revised*).

material from remaining on the sides of the flask out of contact with the acid. Continue boiling for exactly 30 minutes. Remove the flask and filter through fine linen (about 18 threads to a centimetre) held in a funnel, and wash with boiling water until the washings are no longer acid to litmus. Heat some quantity of sodium hydroxide solution to boiling under a reflux condenser. Wash the residue on the linen into the flask with 200 ml of the boiling sodium hydroxide solution. Immediately connect the flask with the reflux condenser and boil for exactly 30 minutes. Remove the flask and immediately filter through the filtering cloth. Thoroughly wash the residue with boiling water and transfer to a Gooch crucible prepared with a thin but compact layer of the ignited asbestos (**F-1.6**). Wash the residue thoroughly first with hot water and then with about 15 ml of ethylalcohol, 95 percent by volume. Dry the Gooch crucible and contents at 105°C in an air-oven to constant weight. Cool and weigh. Incinerate the contents of the Gooch crucible in a muffle furnace at 600 ± 20°C until all the carbonaceous matter is burnt. Cool the Gooch crucible containing the ash in a desiccator and weigh.

F-3. CALCULATION

F-3.1 Crude fibre (on moisture, fat, sugar and milk free basis), percent by mass

$$= \frac{100\ (M_1 - M_2)}{M_3}$$

where

M_1 = mass in g of Gooch crucible and contents before ashing,

M_2 = mass in g of Gooch crucible containing asbestos and ash, and

M_3 = mass in g of the residue taken for the test (**F-2.2**).

APPENDIX G

[*Table* 1, *Item* (viii)]

DETERMINATION OF PARTICLE SIZE

G-1. APPARATUS

G-1.1 Sieve — The sieve comprises a 7·5 cm diameter hollow cylinder of tinned copper 6 cm high to which is a long hoop. The base of the sieve is flanged and covered with standard wire gauze, 150 mesh, and soldered all round the edge. A metal ring is soldered on the lower side of the gauge to protect the sieve when standing on the bench.

G-1.2 Desiccator — 20 cm diameter approximately with tight lids.

G-2. REAGENT

G-2.1 Petroleum Ether — Boiling range 60-80°C.

G-3. PROCEDURE

G-3.1 Place 25 g of liquor in the sieve and lower gently into the desiccator containing petroleum ether, at an angle of 30° to the horizontal. Cover the desiccator and gently rotate it avoiding splashing of ether with material passed through the sieve to come back into it. When the sieve is somewhat less than a quarter full, slightly withdraw it so that about half the gauge is below the surface of the petroleum ether in the vessel. When the volume in the sieve has been reduced appreciably, lower the sieve until it is again somewhat less than a quarter full and drain as before. Repeat the process for 5 minutes, maintaining the gentle swirling motion throughout.

Transfer the sieve to the second desiccator containing clean petroleum ether. Washdown any liquor clinging to the walls using a wash bottle of petroleum ether. Complete sieving by repeatedly dipping and draining to acheive appearance of appreciably coarse cocoa on the sieve.

Dry the sieve in an air oven for 10 minutes. Transfer the cocoa tailings to a tared dish, using a small brush and weigh.

NOTE — After a number of tests, clean the sieve in a large beaker containing 2 cm layer of boiling 3 N sodium hydroxide. Boil the sieve for about 2 minutes. Wash the sieve thoroughly with water, then with alcohol and dry in an oven.

APPENDIX H

(Clause 4.1)

SAMPLING OF COCOA MASS

H-1. GENERAL REQUIREMENTS OF SAMPLING

H-1.0 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

H-1.1 Samples shall be taken in a protected place not exposed to dampair, dust or soot.

H-1.2 The sampling instrument shall be clean and dry when used.

H-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and containers for samples from adventitious contamination.

H-1.4 The samples shall be placed in clean and dry glass containers. The sample containers shall be of such a size that they are almost completely filled by the sample.

H-1.5 Each container shall be sealed air-tight after filling and marked with full details of sampling, batch or code number, name of the manufacturer and other important particulars of the consignment.

H-2. SCALE OF SAMPLING

H-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot.

H-2.1.1 From each lot, samples shall be tested to ascertain the conformity of the material.

H-2.2 The number of containers to be selected randomly. Selection shall depend on the size N of the lot and shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

Lot Size N	Sample Size n
5 to 100	3
101 to 300	5
301 to 500	7
501 and above	9

H-3. PREPARATION OF TEST SAMPLES AND REFEREE SAMPLE

H-3.1 About 250 g of the material shall be taken from each of the selected containers after melting the contents at 55°C and mixing them thoroughly with a sampling tube.

H-3.2 Preparation of Composite Sample — Equal quantities of material (about 100 g) shall be taken from the sample as obtained in **H-3.1** and mixed thoroughly to constitute a composite sample of size 300 g. This sample shall be divided into three equal parts, one for the purchaser, one for the vendor and the third one for the referee. The test samples so obtained shall be transferred immediately to thoroughly clean and dry containers and sealed air-tight. These shall be labelled with particulars of sampling given in **H-1.5**.

H-3.3 Preparation of Individual Sample — The remaining portions of the material from each of the selected containers (after the required quantity needed for formation of the composite sample) shall be divided into three equal parts. These parts shall be immediately transferred to thoroughly dried containers which are then sealed air-tight and labelled with all the particulars given in **G-1.5.**

H-3.4 Referee Samples — Referee samples shall consist of the composite sample (*see* **H-3.2**) and a set of individual test samples (*see* **H-3.3**) marked for this purpose and shall bear the seals of the purchaser and the vendor. These shall be kept at a place agreed to between the two.

H-4. NUMBER OF TESTS

H-4.1 The test for the description, total fat, total ash, acid insoluble ash, alkalinity of ash, and crude fibre shall be conducted on the individual samples as obtained in **H-3.3.**

H-4.2 The test for remaining characteristics shall be made in the composite sample as prepared under **H-3.2.**

H-5. CRITERIA FOR CONFORMITY

H-5.1 A lot shall be declared as conforming to the requirements of this standard if **H-5.1.1** and **H-5.1.2** are satisfied.

H-5.1.1 Each of the test results obtained for various characteristics from individual testing (*see* **H-4**) shall satisfy the corresponding requirements as given in 2.

H-5.1.2 The test results on the composite sample for the characteristics specified in Table 1 shall satisfy the corresponding requirements given in Table 1.

INDIAN STANDARDS

ON

COCOA PRODUCTS

IS :

1163-1971 Chocolate (*first revision*)
1164-1986 Cocoa powder (*third revision*)
1263-1986 Cocoa butter (*third revision*)
6762-1986 Drinking chocolate (*second revision*) (Sweetened Cocoa powder)
8832-1978 Method of cut test for cocoa beans
8833-1978 Method for determination of moisture content cocoa beans
8865-1978 Cocoa beans
10017-1981 Code of practice for construction of cocoa beans storage structures
11730-1986 Glossary of terms for cocoa, chocolate and its products
11923-1986 Cocoa Mass
11924-1986 Chocolate coating